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Electroformed Nanocrystalline Coatings An Advanced Alternative to Hard-Chrome Electroplating PP-1152

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- Technical Objective
- SERDP Program Overview
- Background
- Results Since San Diego Meeting
- Remaining Actions

Technical Objective

- **Develop an environmentally benign advanced nanocrystalline Co-based coating technology that:**
  - **Is compatible with conventional electroplating infrastructure**
  - **Will produce coatings that meet or exceed the overall performance of hard chrome (hardness, wear, fatigue, corrosion, and thermal stability)**
  - **Has costs similar to or less than life-cycle cost of existing hard chrome electroplating processes**
  - **Will be applied to non-line-of-sight surfaces**
- **Cobalt alloy selection**
  - **Mechanical properties**
  - **High plating efficiency**
  - **No constituents on EPA or AFMC lists of hazardous materials**
  - **Longer term view**

Program Overview

Three Phases

- **Phase I Technology Viability Assessment**
  - **Completed**
- **Phase II Coating Optimization**
  - **Completed**
- **Phase III Extension to Complex ID Shapes**
  - **Completed**

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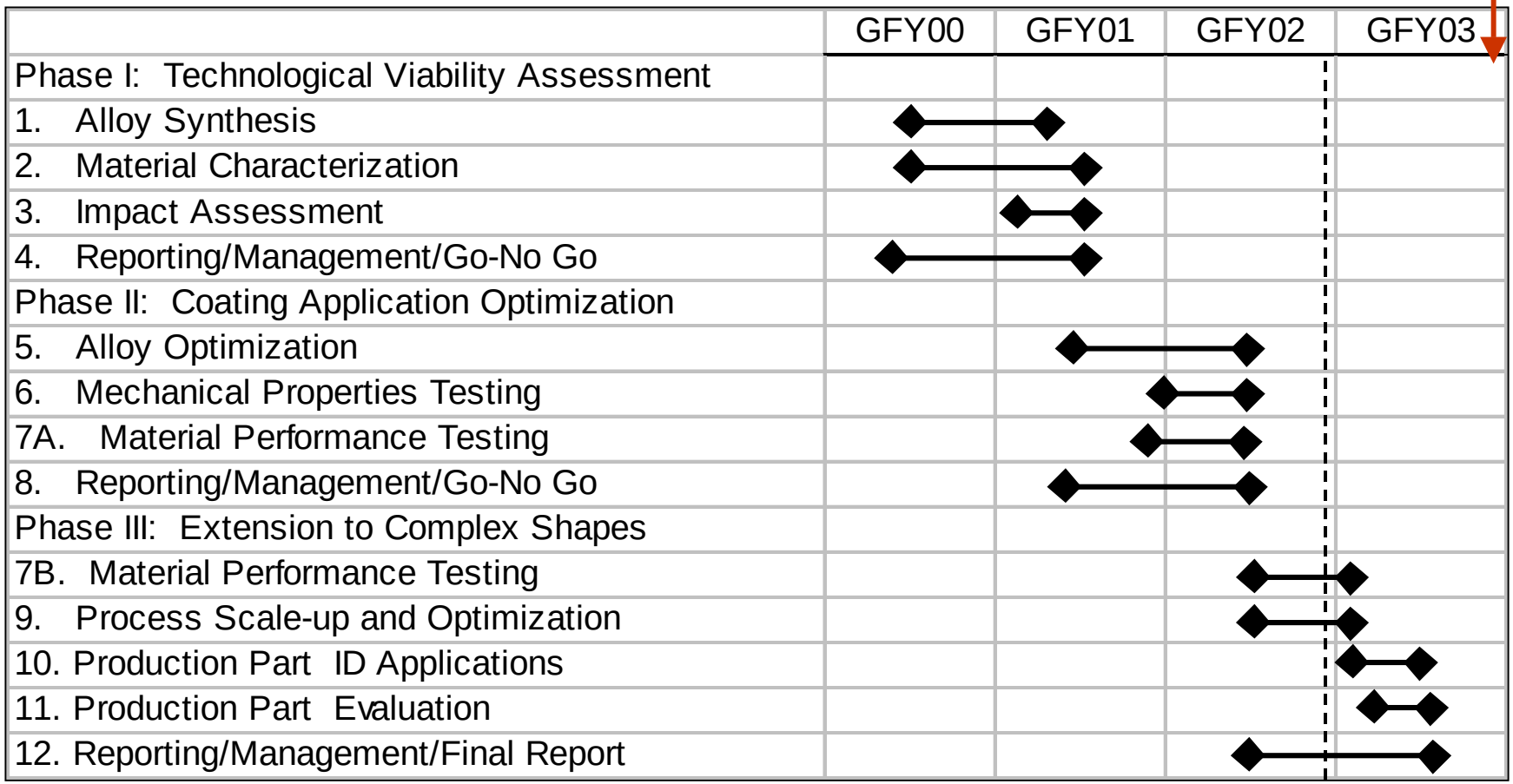
Program Plan

Integran Technologies



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Background

Integran Technologies

- **Synthesized Co-P, Co-Mo and Co-Fe nano alloys**
- **Synthesized and Optimized Co-Fe,Co-Fe-P, Co-Fe-Zn and Co-Fe-Zn-P nano alloys**
- **Optimized Co-P alloy**
  - **Cobalt chloride/ortho-phosphoric/phosphorous acid bath**
  - **Plating efficiency >90%**
  - **Grain size 12-15 nm**
  - **As-deposited hardness 700 VHN**
  - **Deposition rate 2-8 mills/hr**
  - **Precipitation hardenable**
  - **Good salt spray results**
  - **High Taber wear results (CS 17)**
  - **Good pin-on-disk results**

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Activities Since San Diego

- Surface Roughness Study
- Final hydrogen embrittlement tests completed
- Final fatigue tests completed
- Representative ID geometries plated

Surface Roughness Study

- Nano Co 2-3wt% coatings onto mild steel coupons (2” x 3”)
Standard Co-P bath and conditions
50, 100 and 150µm

- Mild Steel test samples were mechanically ground and/or polished with several grit SiC abrasive paper (60, 240 and 1000 grit) to achieve different starting surface roughness

- Surface roughness was measured before and after plating using a stylus profilometer

Surface Roughness

Surface Finish	Surface Roughness, Ra (µm)			
	Before Plating	After Plating		
		50µm Thickness	100µm Thickness	150µm Thickness
60 grit	0.94	0.87	0.88	0.76
240 grit	0.25	0.22	0.22	-
1000 grit	0.11	0.09	0.08	0.09

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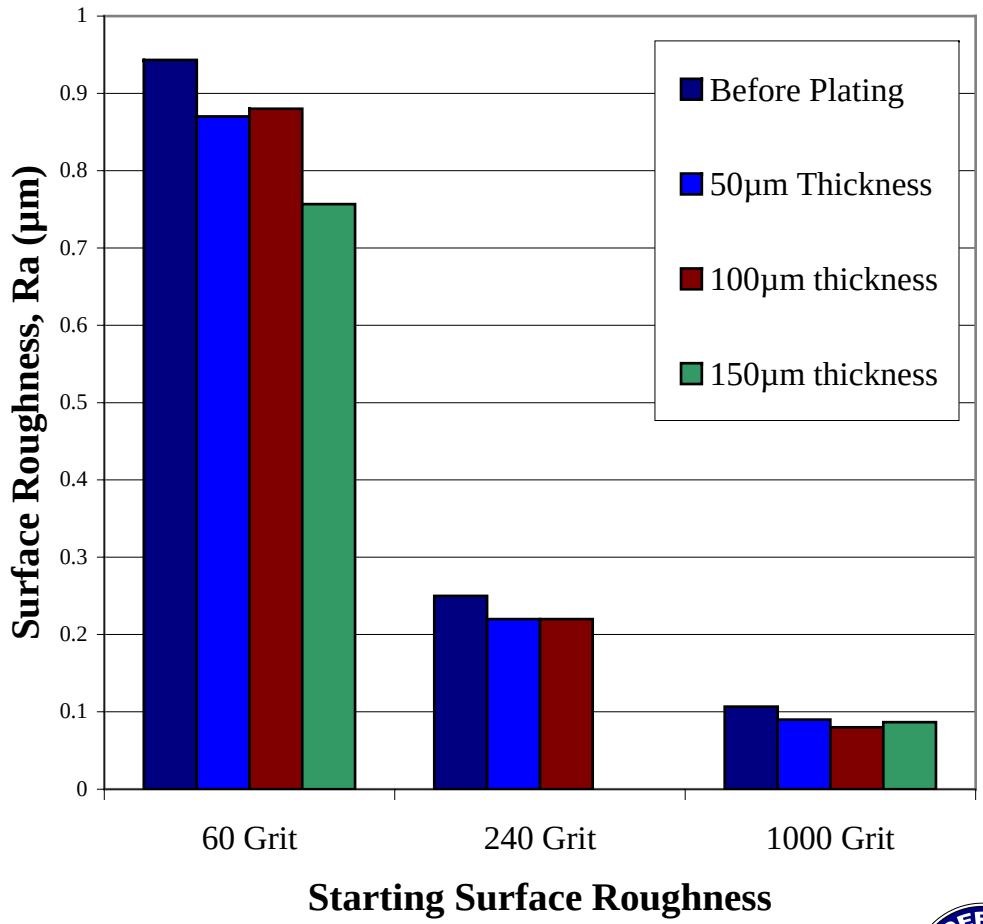
Surface Roughness

Integran™ Technologies

- **Nano Co-P coatings do not significantly alter the starting Ra of the substrate**
- **In some cases the surface roughness slightly improves with increasing coating thickness**



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Fatigue Test Matrix

Integran Technologies

- ASTM E466 – Force Controlled Fatigue at MTI
- Hourglass specimens with 0.25-inch gauge diameter
- 4340 HT (avg ~46 Rc)
- R= -1, Air

Coating	Batch	Peen	Stress Level 110 ksi	125 ksi	150 ksi	175 ksi
Bare	1	No	✔	✔	✔	✔
	1	Yes	✔	✔	✔	✔
Hard Chrome	2	No	✔	-	✔	-
	2	Yes	✔	✔	✔	✔
Nano Co	1	Yes	-	✔	-	✔
Nano Co 2-3wt%P	2	No	✔	-	✔	-
	1	Yes	-	✔	-	✔
	2	Yes	✔	✔	✔	✔
Nano Co 4-5wt%P	1	Yes	-	✔	-	✔
Co 15-20wt%P	2	Yes	✔	-	✔	-

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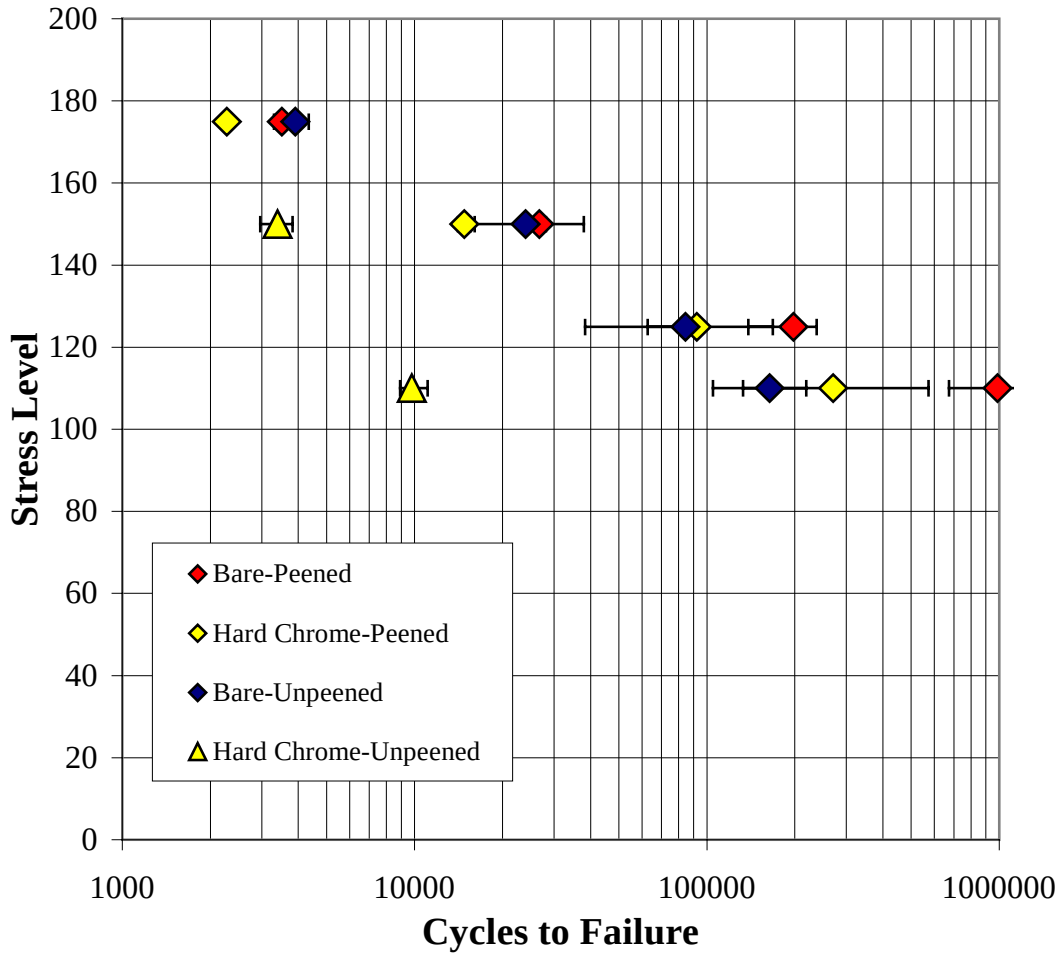
Fatigue Results

Bare and EHC Fatigue Specimens in the Peened and Un-peened Condition



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Fatigue Results

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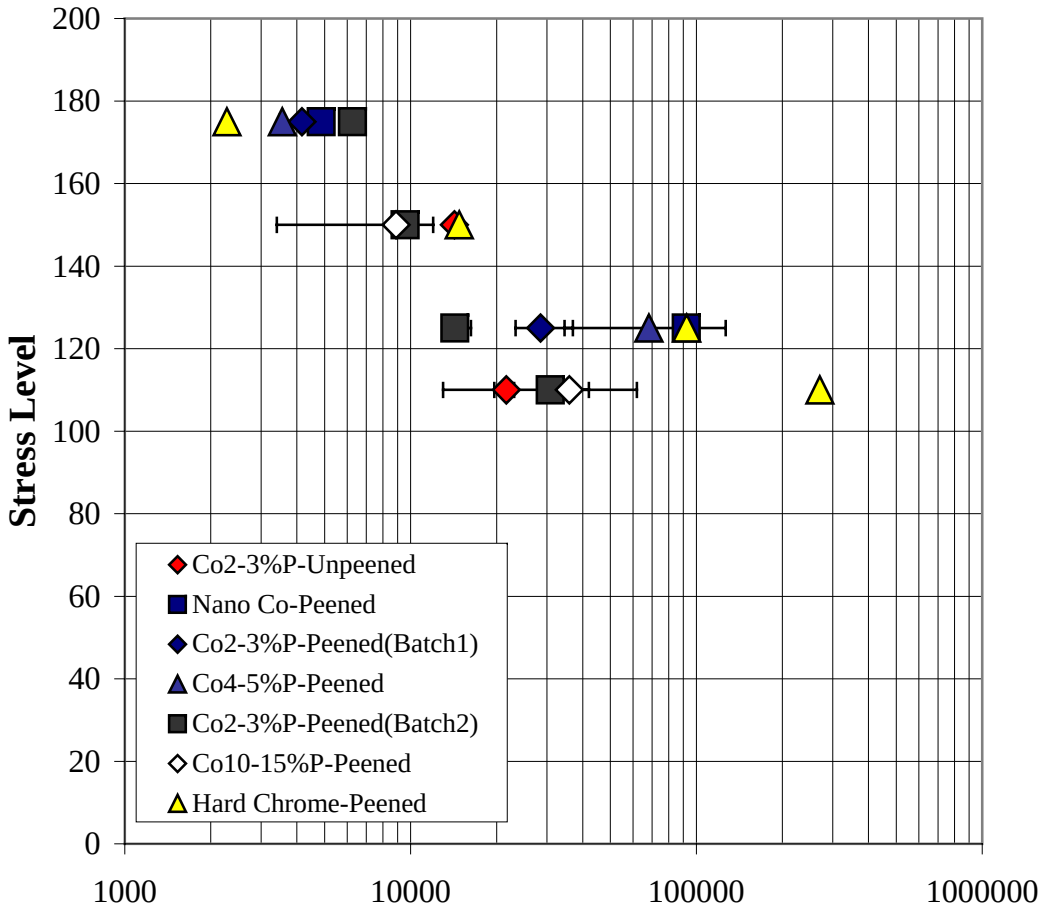
Nano Co-P alloy Fatigue Specimens in the peened and un-peened condition along with hard chrome coated specimens for reference



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Fatigue Test Observations

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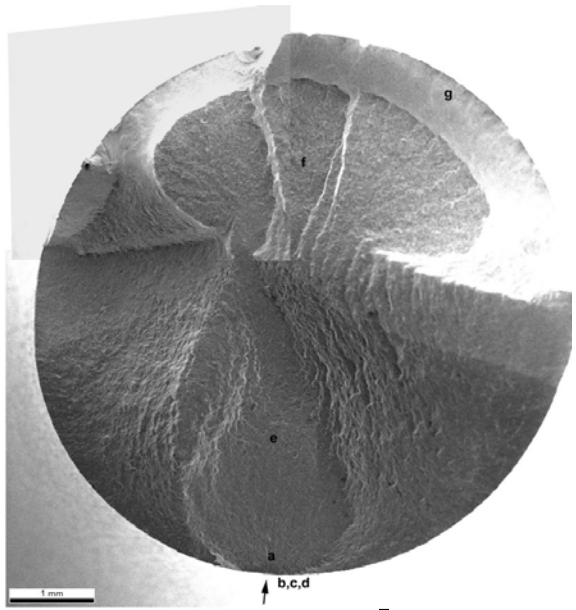
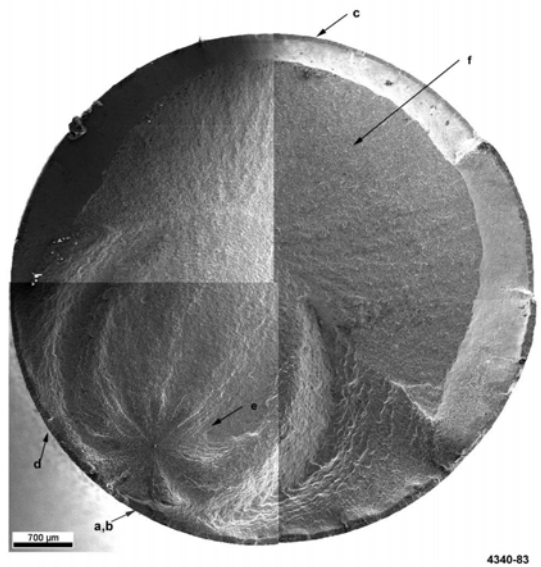
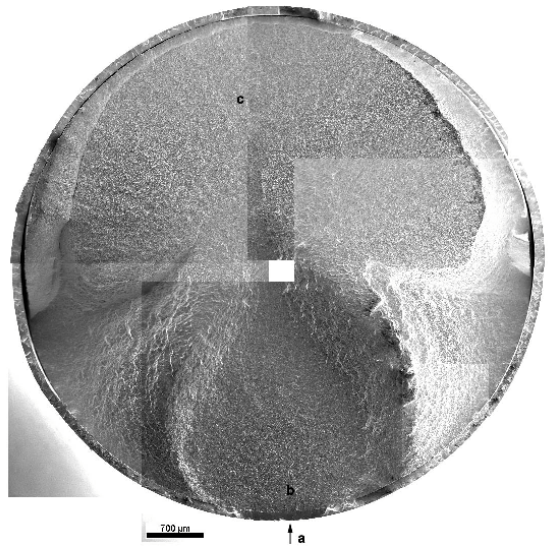


Uncoated



Hard Chrome



Nano Co 2-3wt%

- **Similar fracture face morphology in all cases**
- **No obvious cause of fatigue debit from fracture face analysis**

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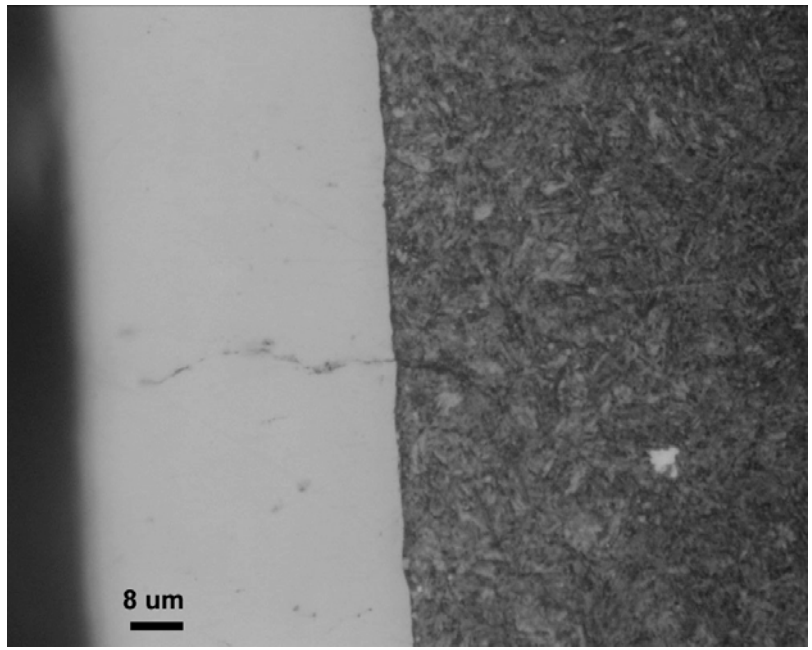
Fatigue Test Observations

Metallographic Cross-Sectional Analysis

Hard Chrome

σ=110ksi cycles = 572,405

- **Very smooth interface**
- **Microcracks from coating extend into substrate**



Hard Chrome

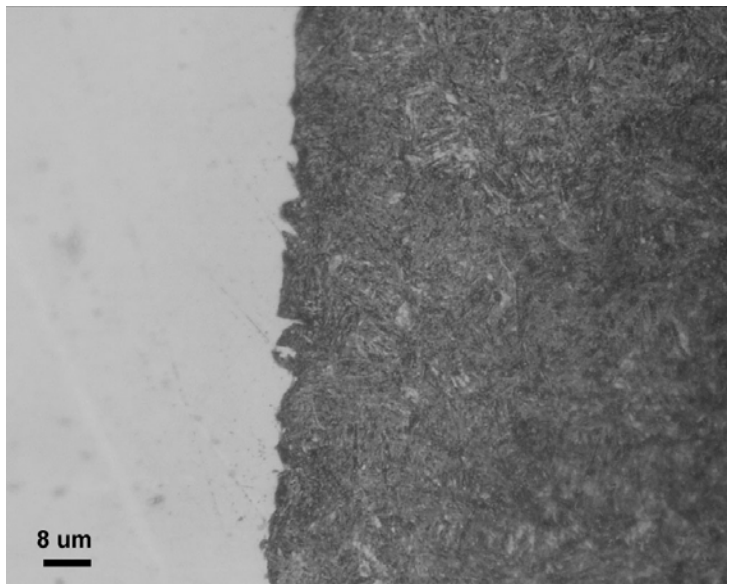
Fatigue Test Observations

Metallographic Cross-Sectional Analysis

Nano Co 2-3wt%P

σ=110ksi cycles = 61,908

- **Very rough interface**
- **No fatigue cracks found extending from coating into substrate**
- **Some trapped Al_2O_3 particles found at coating/substrate interface**



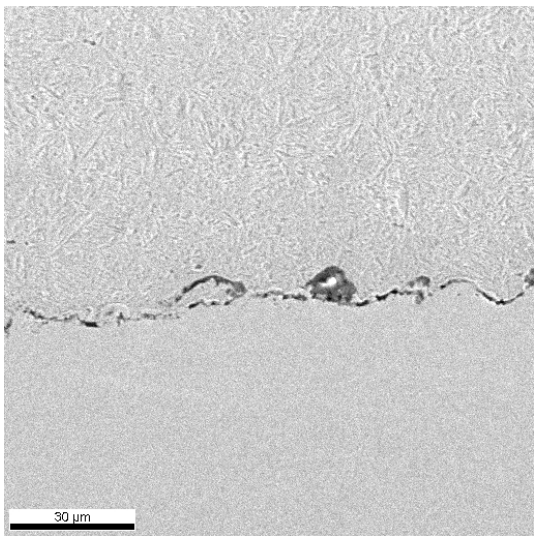
Nano Co 2-3wt%P

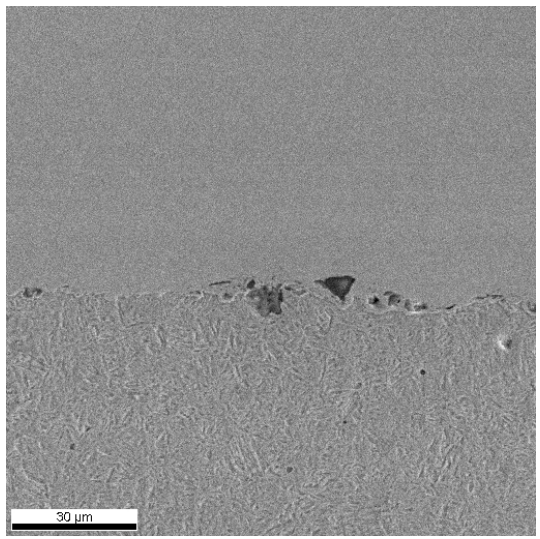
Fatigue Test Observations

Metallographic Cross-Sectional Analysis

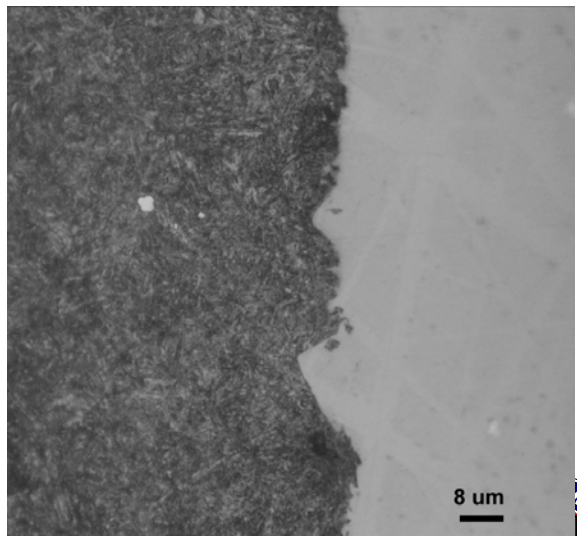
- **Very rough interface in all Nano coated samples**
- **Trapped Al_2O_3 particles from sand blasting found at coating/substrate interface in most Nano coated sample**

Nano Co 4-5wt%P





Nano Co 15-20wt%P

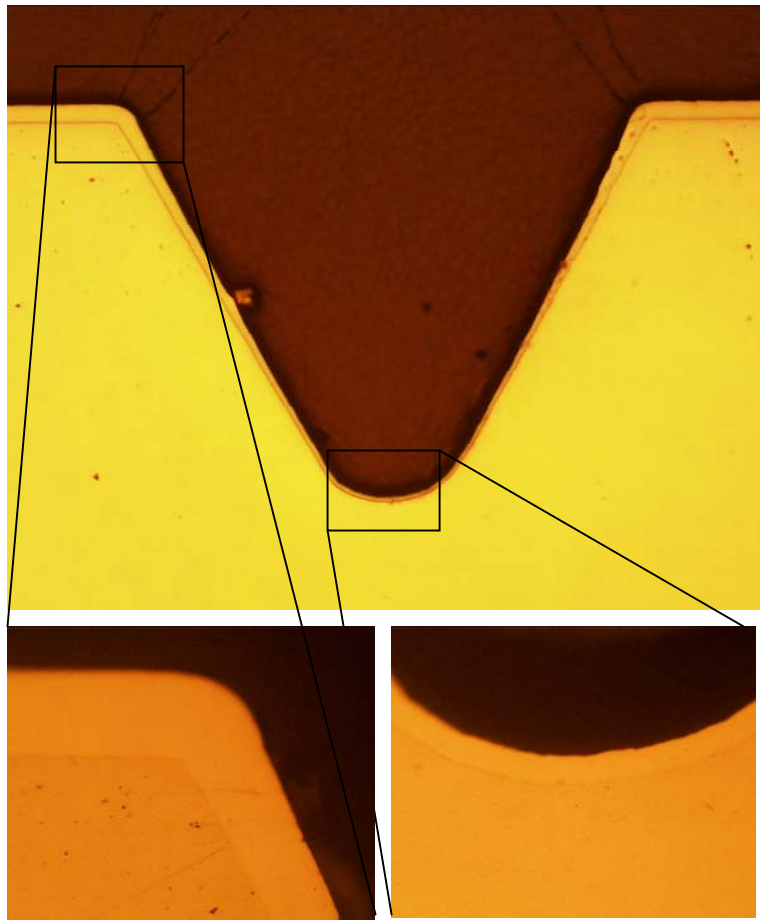


Fatigue Test Observations

- **Debit evident for Hard Chrome at all Stress Levels**
  - **Debit much larger for Un-peened samples**
  - **Failures initiate from microcracks in the coating extending into the substrate**
- **Debit evident for all Co-P alloys at Stress levels of 110, 125, 150 ksi, but not for 175ksi.**
  - **Peened and un-peened fatigue specimens coated with Nano Co-P had similar fatigue results.**
  - **Failures likely initiate at very rough surface and/or at trapped Al_2O_3 particles from sand blasting cleaning step.**

H_2 Embrittlement Results

- **F-519 specimens**
- **Carefully selected specimens based on hardness and visual inpection**
- **Tested Hard Chrome with no Bake and baked per Mil-Spec-1501C**
- **Tested Nano Co 2-3 wt% P with no bake and baked for 12 and 36hours at 375°F (191°C)**



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H_2 Embrittlement – Hard Chrome

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- ASTM Type 1a Specimens -.003” coating thickness
- Acceptance criteria: 4 > 200 hrs @ 75% NFS or 3 > 90% NFS for 1 hr

Coating	Bake Condition	Hours at 75% NFS	Failure % of NFS	Pass / Fail
Electrolytic Hard Chrome	**No Bake**	0.5	75	Fail
		1.0	75	Fail
		Not Tested	-	-
		Not Tested	-	-
	24 hours, 191°C	200	90	Pass
		211	90	Pass
		233	90	Pass
		213	90	Pass

H_2 Embrittlement – Nano Co-P

Integran Technologies

Coating	Bake Condition	Hours at 75% NFS	Failure % of NFS	Pass / Fail
Nano Co 2-3wt%P	No Bake	205	90	Pass
		208	90	Pass
		203	75	Pass
		205	90	Pass
	12 hours, 191°C	234	85	Pass
		212	90	Pass
		208	90	Pass
		>200	>75	Pass
	36 hours, 191°C	212	90	Pass
		209	90	Pass
		242	85	Pass
		208	90	Pass

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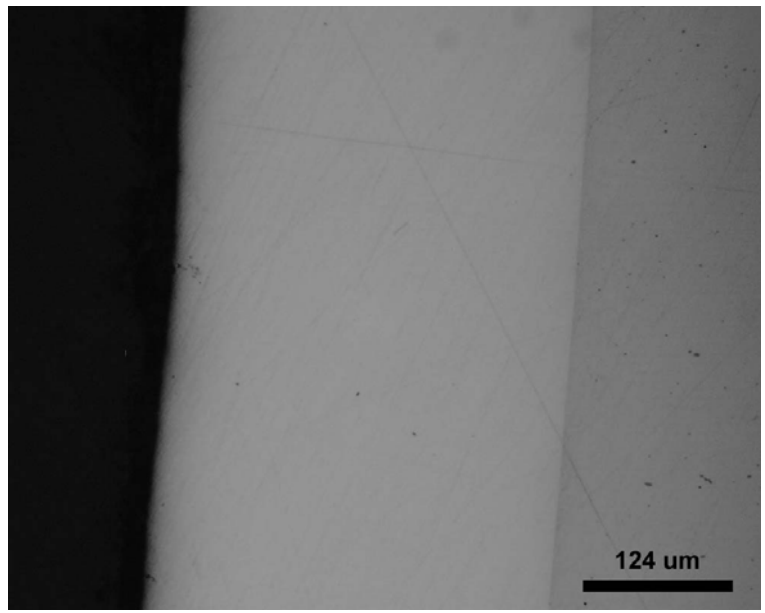
Application to ID Surfaces

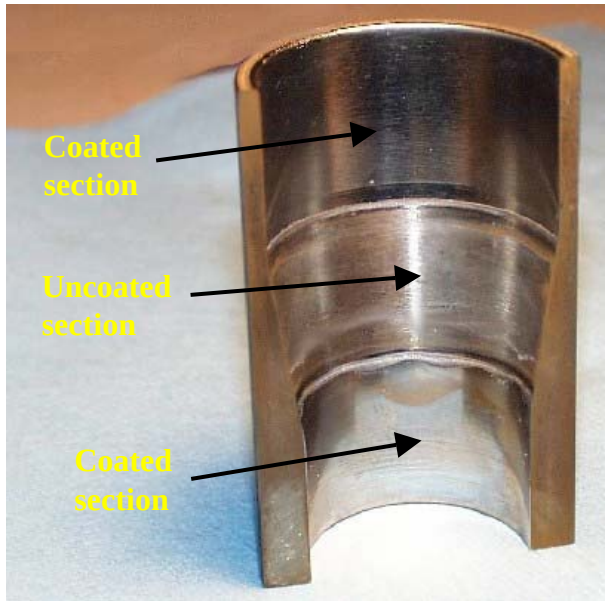
- **Mockups utilized**
  - **Blind and through cylinders**
  - **Pins**
  - **External lugs**
- **Anode design study**
  - **Small ID surfaces**
    - **Non-consumable graphite anode**
  - **Large ID surfaces**
    - **Consumable Co anode**



Optical Micrograph - 13 mil Nano Co 2-3wt%P coating on 1” ID

Small ID Surfaces

Stepped ID

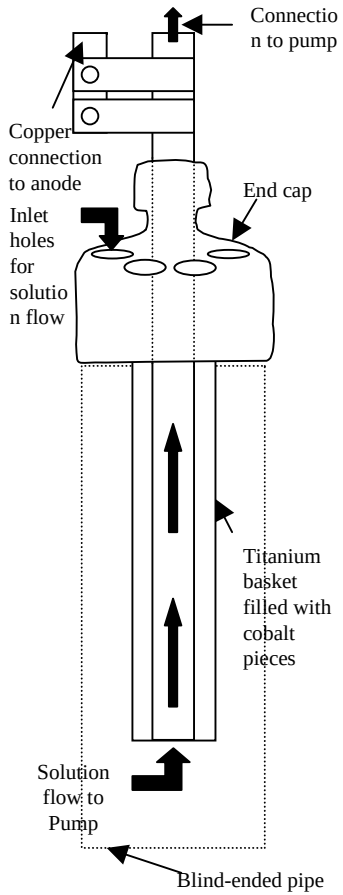


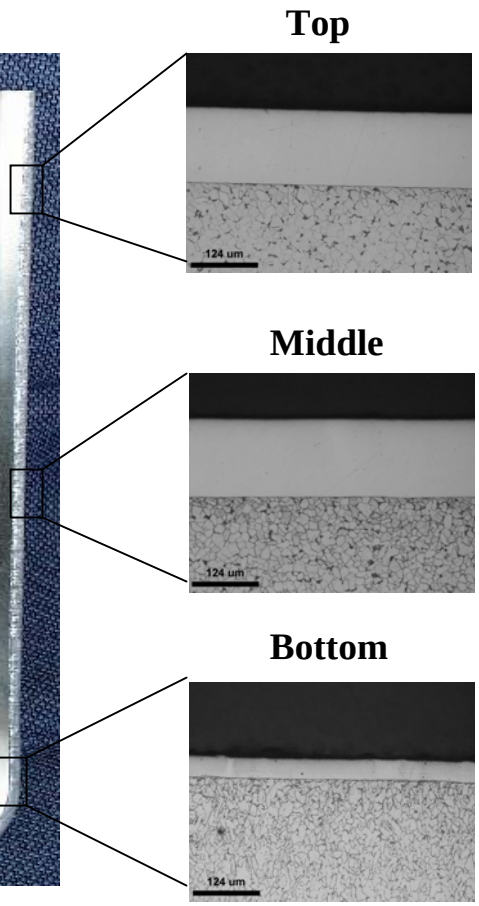
- Stepped graphite anode
- Plastic plug used to mask uncoated section, with holes to allow for flow

- Uniform coating thickness on the two ID surfaces

Large ID Blind Hole

Plating Assembly





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Final Demonstration Part

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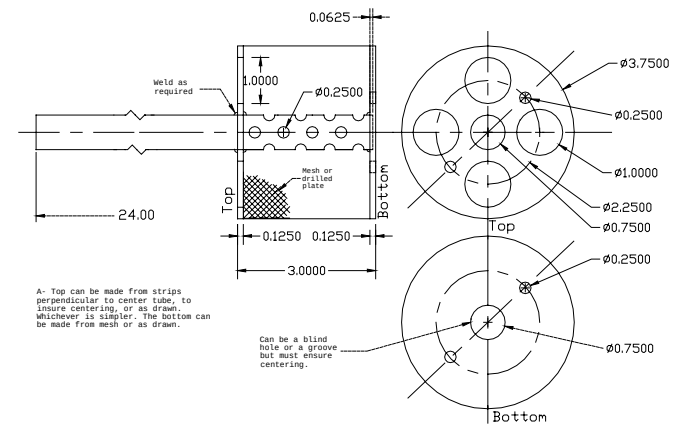
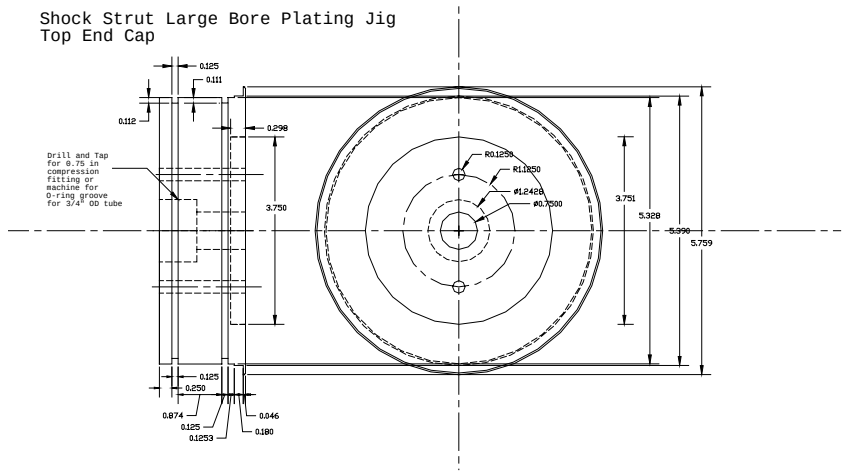
The shock strut of a landing gear component (provided courtesy of Messy-Dowty Aerospace)

•Designed anode basket and accompanying brace to centre anode in component and to mask edges to produce the required coating fade-out at edges





Final Demonstration Part

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The shock strut of a landing gear component (provided courtesy of Messy-Dowty Aerospace)

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Process Data Summary

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	Nanocrystalline Co-P Alloy	Hard Chrome
Bath Chemsitry	Co 2-5wt%P ($CoCl_2$ / H_3PO_4 / H_3PO_3)	Cr (CrO_3 / SO_4^{2-})
Efficiency	85-95%	15-35%
Deposition Rate	Up to 8 mil per hour	Up to 1.6 mil per hour
Thickness	Demonstrated up to 0.020”	Typically < .005”
As-Deposited Appearance	Pit / Pore Free	Microcracked
Microstructure	Nanocrystalline (avg. g.s. = 8 -15nm)	-
Relative Process Cost	1.3	1.0
Emission Analysis	Below OSHA limits	Cr^{6+}

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Property Data Summary

Integran Technologies

		Nanocrystalline Co-P	Hard Chrome
Hardness	*As-Deposited*	600-700 VHN	800-1200 VHN
	HT @ 250°C	700-800 VHN	-
	HT @ 400°C	1000-1200 VHN	-
Ductility		2 – 7 % Elongation	<.1%
Thermal Stability		400°C	-
Wear	*Abrasive (Taber)*	27 mg / 1000 cycles (CS-17) 11 mg / 1000 cycles (CS-10)	3.2 mg / 1000 cycles (CS-17) 1.0 mg / 1000 cycles (CS-10)
	Adhesive (Pin-on-disk)	5-6 x 10^{-6} mm^3/Nm (Alumina Ball on Nano Co-P Disk)	9-11 x 10^{-6} mm^3/Nm (Alumina Ball on Cr Disk)
	Coefficient of Friction	0.5	0.7
Corrosion	Salt Spray	Protection Rating 7 @ 1000 hrs	Protection Rating 2 @ 1000 hrs
	Poteniodynamic	.07 - .15 mpy	.02 mpy
Internal Stress		10-15 ksi (Tensile)	Cracked – Exceeds cohesive strength
Hydrogen Embrittlement		No – No Bake Required	Yes – min bake 14 hrs
Fatigue		Additional tests needed	Fatigue Debit

Remaining Actions

- **Submit final report**
- **Additional Fatigue Testing?**
- **Start ESTCP Program**